
क्विनालफोस, तकनीकी — विशिष्टि
(दूसरा पुनरीक्षण)

Quinalphos, Technical —
Specification
(Second Revision)

ICS 65.100.10

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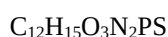
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FOREWORD

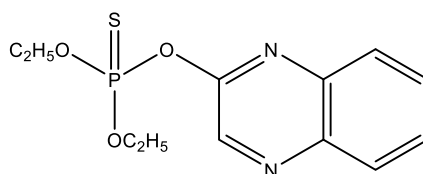
This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Quinalphos is the accepted common name by the International Organization for Standardization (ISO) for *O,O*-quinoxalin-2- γ -phosphorothioate. The empirical and structural formulae and molecular mass are as given below:

Empirical Formula



Structural Formula



Molecular Mass

298.3

This standard was first published in 1976 and subsequently, an amendment was issued to it. In the first revision issued in 1984, a thin layer chromatography method followed by ultraviolet absorption procedure for determination of purity of quinalphos was incorporated in addition to the volumetric method. Three amendments issued to the standard and through the third amendment high performance liquid chromatography (HPLC) and gas liquid chromatography (GLC) methods were included for determination of quinalphos content in addition to the existing methods.

In this revision, thin layer chromatography method followed by ultraviolet absorption procedure for determination of purity of quinalphos has been removed as the same is an outdated method and it does not give the accurate results. The standard has also been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates three amendments issued to this standard.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968, and the rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

The composition of the committee responsible for the formulation of this standard listed in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
QUINALPHOS, TECHNICAL — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for quinalphos, technical.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
IS 6940 : 1982	Methods of test for pesticides and their formulations (<i>first</i>
IS 8190 (Part 2): 1988	Requirements for packing of pesticides: Part 2 Liquid pesticides (second revision)
IS 10946 : 1996	Methods of sampling for technical grade pesticides

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of a reddish brown to dark brown clear liquid free from extraneous matter.

3.2 The material shall also comply with the requirement specified in Table 1.

4 PACKING

The material shall be packed as per requirements given in IS 8190 (Part 2).

5 MARKING

5.1 The container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name and address of the manufacturer;
- c) Batch number;
- d) Date of manufacture;
- e) Date of expiry;
- f) Net quantity;
- g) Nominal quinalphos content, percent (*m/m*); and
- h) Cautionary notice as worded in the *Insecticides Act, 1968*, and rules framed thereunder; and
- j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules, 2011*.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn in accordance with IS 10946.

7 TESTS

7.1 Tests shall be carried out as prescribed in co1 (4) of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of an analysis.

Table 1 Requirements for Quinalphos, Technical
(Clause 3.2)

Sl No.	Characteristics	Requirement	Method of Test, Ref To
(1)	(2)	(3)	(4)
i)	Quinalphos content, percent by mas, <i>Min</i>	70.0	Annex A
ii)	Moisture content, percent by mas, <i>Max</i>	0.1	IS 6940
iii)	Relative density at 27/27 °C, <i>Min</i>	1.04	IS 6940
iv)	Material insoluble in acetone, percent by mass, <i>Max</i>	0.1	IS 6940
v)	Acidity (as H ₂ SO ₄) percent by mass, <i>Max</i>	0.4	IS 6940

ANNEX A

[Table 1, Sl. No. (i)]

DETERMINATION OF QUINALPHOS CONTENT

A-1 GENERAL

Two methods, namely, high performance liquid chromatography (HPLC) or gas liquid chromatography (GLC) may be used for determination of quinalphos content. In addition, a volumetric method may be used only by a manufacturer solely for production quality control purposes. The volumetric method shall not be used by an independent testing laboratory.

A-2 HPLC METHOD

A-2.1 Principle

Equal volumes of solutions containing known amounts of quinalphos standard and internal standard are injected into the HPLC unit fitted with a UV detector and the response factor (R.F.) is determined. Same volume of solution containing known amounts of the sample and internal standard is injected subsequently into the HPLC unit and quantity of quinalphos is computed by the standard relationship.

A-2.2 Apparatus

A-2.2.1 High Performance Liquid Chromatograph

Equipped with a printer-plotter-cum-integrator and UV detector. The suggestive operative parameters are given below, which can be varied provided standardization is done:

Column	Stainless steel, 25 cm length and 4.6 mm ID; packed with silica of 10 µm particle size
Detector wavelength	305 nm
Solvent system	Carbon tetrachloride, 85 percent (v/v) and Chloroform, 15 percent (v/v)
Solvent flow rate	1.0 ml/min
Sample size	10 µl

A-2.2.2 Micro-Syringe

A-2.3 Reagents

A-2.3.1 Internal Standard Solution — One gram meta phenoxy benzaldehyde (MPB), AR grade, dissolved in 100 ml of the solvent system as described above (see A-2.2.1) in a 100 ml volumetric flask. The final solution will contain 10 mg/ml of internal standard (MPB).

A-2.3.2 Quinalphos Standard Solution — Dissolve 0.1 g quinalphos, reference standard in a 100 ml volumetric flask in the solvent system. Then add 10 ml internal standard solution and finally make up the volume with the solvent system. The final solution will contain 1 000 parts per million of quinalphos and MPB (Solution I).

A-2.3.3 Sample Solution — Dissolve in a 100 ml volumetric flask 0.12 g of quinalphos, technical or 0.4 g of quinalphos 25 percent EC sample in the solvent system. Add 10 ml internal standard solution and finally make up the volume with the solvent system.

A-2.4 Estimation

A-2.4.1 Introduce simultaneously 10 µl each of the internal standard solution and quinalphos standard solution into the HPLC unit and calculate the response factor (R.F.)

$$R.F. = \frac{\text{Peak area for quinalphos}}{\text{Peak area for MPB}} \times \frac{\text{Mass of MPB}}{\text{Mass of quinalphos}} \times$$

Percent purity of quinalphos standard

A-2.4.2 Introduce 10 µl of sample solution into the HPLC unit, measure the peak areas for the internal standard and quinalphos in the sample solution. Calculate the quinalphos content.

A-2.5 Calculation

Quinalphos content, percent by mass =

$$\frac{\text{Peak area for quinalphos}}{\text{Peak area for MPB}} \times \frac{\text{Mass of MPB}}{\text{Mass of sample}} \times 100$$

A-3 GLC METHOD

A-3.1 Principle

The quinalphos content is determined using an internal standard on a GLC equipped with a flame ionization detector (FID) and integrator.

A-3.2 Apparatus

A-3.2.1 Gas Liquid Chromatograph

A gas chromatograph equipped with a FID detector is operated under the following suggested parameters. These parameters may be varied as per available facilities provided standardization is done:

Column	Glass, 200 cm long and 3 mm i.d. packed with 3 percent OV-17 on 80 mesh gas chrome Q to 100 mesh gas chrome Q
Column oven temperature	210 °C
Injection port temperature	250 °C
Detector temperature	250 °C
Gas flow rate	Nitrogen, 30 ml/mm

A-3.2.2 Micro-Syringe — 2 µl

A-3.3 Reagents

A-3.3.1 Quinalphos Reference Standard — of known purity

A-3.3.2 Acetone/Toluene

A-3.3.3 Internal Standard — Di-n-butyl phthalate

A-3.4 Procedure

A-3.4.1 Preparation of Internal Standard Solution

Weigh 1.5 g of di-n-butyl phthalate in a 50 ml volumetric flask and make up to volume using acetone/toluene. This will give a solution containing 30 mg/ml of internal standard.

A-3.4.2 Preparation of Reference Standard Solution

Weigh equivalent to 0.2 g of quinalphos in a beaker. Add 5 ml of internal standard solution (see **A-3.4.1**). Shake well to dissolve and transfer carefully to a 25-ml volumetric flask and make up the volume with acetone/toluene.

A-3.4.3 Preparation of Sample Solution

Weigh a sample containing equivalent to 0.2 g of quinalphos and proceed as described in **A-3.4.2**.

A-3.4.4 Estimation

Inject 2 µl of the standard into the GLC and, if necessary, adjust the parameters and the column injected to give a complete separation within a reasonable time and peak heights half to three-fourths full scale. Proceed with determination at least reinjecting each of standard and sample solution in random order.

A-3.4.5 Measure the peak heights of areas of quinalphos and internal standard solution. Determine the R.F. values for each injected standard and internal standard solution.

A-3.5 Calculation

Quinalphos content, percent by mass =

$$\frac{M_{IS} \times A_S}{M_S \times A_{IS}} \times R_f \times 100$$

where

M_{IS} = mass, in g, of the internal standard;

A_S = area of the sample peak;

R_f = response factor = $\frac{M_{IS} \times A_S}{M_S \times A_{IS}}$

M_S = mass, in g, of the sample taken; and

A_{IS} = area of the internal standard peak.

A-4 VOLUMETRIC METHOD

A-4.1 Principle

Quinalphos is decomposed by heating it with hydrogen peroxide and concentrated sulphuric acid, and the phosphate obtained is estimated volumetrically by the molybdate method.

A-4.2 Reagents

A-4.2.1 Nitric Acid — 70 percent

A-4.2.2 Sulphuric Acid — 96 percent

A-4.2.3 Citric Molybdate Reagent — Prepared as in **A-4.2.3.1** and **A-4.2.3.2**

A-4.2.3.1 To 54 g anhydrous molybdic acid, add 200 ml water and 15 g sodium hydroxide. Heat and stir until dissolved.

A-4.2.3.2 Dissolve 60 g citric acid in 250 ml to 300 ml water. Add 140 ml concentrated hydrochloric acid (rd 1.18). To this solution, add solution prepared in **A-4.2.3.1** with stirring. Filter and dilute to one litre. This solution gives blue-green tinge. Add 0.5 percent to 1 percent potassium bromate till the colour becomes yellow.

A-4.2.4 Hydrogen Peroxide — 30 percent (v/v)

A-4.2.5 Mixed Indicator Solution — Prepared by mixing 3 volumes of Solution 1 and 2 volumes of Solution II.

Solution I — Dissolve 0.1 g thymol blue in 2.2 ml of 0.1 N sodium hydroxide and 50 ml alcohol. Dilute to 100 ml with water.

Solution II — Dissolve 0.1 g phenolphthalein in 60 ml alcohol and dilute to 100 ml with water.

A-4.2.6 Quinoline Solution

Heat 60 ml concentrated hydrochloric acid in 350 ml to 400 ml water to 70 °C to 80 °C. To this hot solution add 50 g quinoline, stir and after cooling, dilute to 1 litre.

A-4.2.7 Hydrochloric Acid — concentrated and 0.5 N

A-4.2.8 Sodium Hydroxide Solution — 0.5 N

A-4.3 Procedure

A-4.3.1 Weigh sample to contain 0.5 g quinalphos in a glass ampoule- and introduce it in a 500 ml Kjeldahl digestion flask. Add 20 ml of hydrogen peroxide and cool in ice-cold water. Add 10 ml of sulphuric acid and heat gently under controlled conditions till white fumes of sulphur trioxide come

out. Allow to cool, add 15 ml of nitric acid and heat again to disappearance of brown fumes and appearance of white fumes of sulphur trioxide. Allow to cool, add 20 ml of hydrogen peroxide and heat again till white fumes are given off. Cool and make up to 500 ml.

A-4.3.2 To 100 ml aliquot from **A-4.3.1**, add 50 ml of citric molybdate reagent. Heat to boiling and add very slowly, dropwise, 25 ml of quinoline solution with constant mechanical stirring. Boil for 5 min more, cool and filter the precipitate through a Gooch crucible. Wash with water till free from acid. Dissolve the precipitate in a known excess of sodium hydroxide solution (about 50 ml of 0.5 N). Titrate excess alkali with 0.5 N hydrochloric acid using mixed indicator. Carry out a blank determination on the reagents and subtract from the volume consumed by the sample.

A-4.4 Calculation

$$\text{Quinalphos content, percent by mass} = \frac{v}{m} \times 2.867$$

where

v = volume in ml of standard sodium hydroxide solution; and

m = mass in g of sample taken for the test.

ANNEX B
(Foreword)

COMMITTEE COMPOSITION
Pesticides Sectional Committee, FAD 01

<i>Organization</i>	<i>Representative(s)</i>
Directorate of Plant Protection Quarantine and Storage, Faridabad	DR RAVI PRAKASH (Chairperson)
All India Biotech Association, New Delhi	SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (<i>Alternate</i>)
Central Insecticide Board and Registration Committee, Faridabad	SECRETARY DR VANDANA SETH (<i>Alternate</i>)
Central Insecticide Laboratory, Faridabad	DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (<i>Alternate</i>)
Consumer Guidance Society of India, Mumbai	SHRI SITARAM DIXIT DR M. S. KAMATH (<i>Alternate</i>)
Crop Care Federation of India, New Delhi	DR J. C. MAJUMDAR
Crop Life India, New Delhi	SHRI ASITAVA SEN MS NIRUPAMA SHARMA (<i>Alternate</i>)
CSIR -Indian Institute of Toxicology Research, Lucknow	DR SHEELENDRA P. SINGH
FMC India Pvt Ltd, Bengaluru	SHRI CHIRAG PATEL
Food Safety and Standards Authority of India, New Delhi	ADVISOR (STANDARDS)
IDMA Laboratories Ltd, Chandigarh	DR INDRA RAI
Indian Agricultural Research Institute, New Delhi	DIRECTOR
Indian Institute of Packaging, Mumbai	DR TANWEER ALAM
Indian Pest Control Association, New Delhi	SHRI UDAYAN GHOSH
Institute of Pesticide Formulation Technology, Gurgaon	DR M. VAIRAMANI
Ministry of Agriculture, Department of Agriculture, Chennai	JOINT DIRECTOR OF AGRICULTURE (RES) DEPUTY DIRECTOR LAB (<i>Alternate</i>)
National Centre for Integrated Pest Management, New Delhi	DR SUMITRA ARORA
National Institute of Plant Health Management, Hyderabad	DR MAHESH SAINI MS T. SRIDEVI (<i>Alternate</i>)

<i>Organization</i>	<i>Representative(s)</i>
Pesticide Manufactures and Formulators Association of India (PMFAI), Mumbai	DR ARCHANA SRIVASTAVA DR UDAY KUMAR (<i>Alternate</i>)
Regional Pesticides Testing Laboratory, Chandigarh	SHRI V. VASU
In Personal Capacity (<i>4-6-90/2/8/2, Sri Devi Nilayam Tejaswinagar Coloni, Attapur, Hyderabad – 500048</i>)	SHRI C. V. RAO
In Personal Capacity (<i>263, Sector 28, Faridabad – 121008</i>)	SHRI VIPIN SAINI
BIS Directorate General	SHRIMATI SUNEETI TOTEJA, SCIENTIST ‘E’/DIRECTOR AND HEAD (FOOD AND AGRICULTURE) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]
<i>Member Secretary</i> SHRI KULDEEP MITTAL SCIENTIST ‘B’/ASSISTANT DIRECTOR (FOOD AND AGRICULTURE), BIS	

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Amendments Issued Since Publication

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